= 7 Hz, 6 H); ^{13}C NMR (CCl$_4$) 214.6, 98.9 (d, *J* = 51.3 Hz), 72.6 (d, *J* = 299.1 Hz), 62.1 (d, *J* = 4.88 Hz), 46.8, 37.1, 28.8, 19.2 (d, *J* = 4.88 Hz), 16.2 (d, *J* = 6.1 Hz); ^{31}P NMR (CCl$_4$) –8.0; MS, *m/e* (relative intensity) 258 (11), 229 (15), 201 (11), 161 (99). Anal. Calcd for C$_{12}$H$_{19}$O$_4$: C, 56.00; H, 7.44. Found: C, 55.77; H, 7.43.

General Hydrolysis Procedure. Preparation of 8. To solution of 7 (0.183 g, 0.70 mmol), HgSO$_4$ (51.9 mg, 0.18 mmol), and EtOH (2 mL) at 0 °C was added 10% H$_2$SO$_4$ (0.69 mL), and the resultant reaction mixture was stirred at room temperature for 48 h. The solvent was evaporated and the residue washed with CHCl$_3$ (3 × 2 mL), dried through MgSO$_4$, and evaporated to give 0.188 g (98%) of 8: IR (CHCl$_3$) 1732, 1715 cm^{-1}; ^{1}H NMR (CDCl$_3$) 4.1 (m, 4 H), 3.05 (d, *J* = 21 Hz, 2 H), 2.6–1.4 (m, 9 H), 1.2 (t, *J* = 7 Hz, 6 H); ^{13}C NMR (CDCl$_3$) 211, 200.2 (d, *J* = 4.9 Hz), 62.8 (d, *J* = 7.3 Hz), 44.9, 43.8, 42.6 (d, *J* = 126.9 Hz), 37.4, 29.3, 20.9, 16.4 (d, *J* = 6.1 Hz); ^{31}P NMR (CDCl$_3$) +19.6, +31.9 (enol); MS, *m/e* (relative intensity) 276 (15), 248 (10), 220 (55), 179 (39), 152 (82), 125 (57). Anal. Calcd for C$_{12}$H$_{21}$O$_5$P: C, 52.17; H, 7.66. Found: C, 52.08; H, 7.67.

Bicyclo[3.3.0]oct-1-en-3-one (9). To a solution of 8 (29 mg, 0.11 mmol) in benzene/water (2 mL, 1:1, v/v) at room temperature was added a solution of tetrabutylammonium hydroxide (0.3 mL, 0.18 mmol, 40 wt % in water). The resulting mixture was vigorously stirred for 2 h, and the layers separated. The aqueous phase was extracted with Et$_2$O (2 × 2 mL), and the combined organic layers were dried with MgSO$_4$, evaporated, and chromatographed (Et$_2$O) to give 11 mg (90%) of 9: IR (CHCl$_3$) 1700, 1620 cm^{-1}; ^{1}H NMR (CDCl$_3$) 5.9 (s, 1 H), 2.4–3.2 (m, 4 H), 1.6–2.4 (m, 4 H), 0.8–1.6 (m, 1 H); ^{13}C NMR (CDCl$_3$) 210.6, 190.8, 124.9, 46.9, 42.4, 31.2, 26.3, 25.6; MS, *m/e* (relative intensity) 122 (100), 81 (0.5).

Registry No. 1, 110271-58-4; 2, 18669-04-0; 3, 7653-22-7; 4, 110271-59-5; 5 (*n* = 2), 110294-72-9; 5 (*n* = 3), 110271-62-0; 6 (*n* = 2), 110271-61-9; 6 (*n* = 3), 110271-63-1; 7, 110271-60-8; 8, 77861-34-8; 9, 72200-41-0; C(O)CH(CO$_2$Et)CH$_2$CH$_2$CH$_2$, 611-10-9; H$_3$CC(O)CH(CO$_2$Et)CH$_3$, 609-14-3; C(O)C(CO$_2$Et)(CH$_2$C≡CP(O)(OEt)$_2$)CH$_2$CH$_2$CH$_2$, 110271-64-2; C(CO)C(CO$_2$Et)CH$_2$C(O)CH$_2$P(O)(OEt)$_2$)CH$_2$CH$_2$CH$_2$, 110271-65-3; H$_3$CC(O)C(CO$_2$Et)(CH$_2$C≡CP(O)(OEt)$_2$)CH$_3$, 110271-66-4; H$_3$CC(O)C(CO$_2$Et)(CH$_2$C(O)CH$_2$P(O)(OEt)$_2$)CH$_3$, 110271-67-5; cyclopropanone, 5009-27-8; cyclobutanone, 1191-95-3; cyclopentanone, 120-92-3; propargyl alcohol, 107-19-7; ethyl vinyl ether, 109-92-2; diethyl chlorophosphate, 814-49-3.

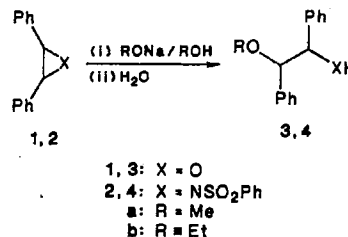**Reactivity Difference of Cis–Trans Pairs: Different Behavior of Stilbene Oxides and Activated Stilbene Imines[1,2]**

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Recently[3,4] the hypothesis was put forward that nucleophilic ring opening of aziridines with a tervalent ni-

(1) Reactions with Aziridines. 43.
(2) Part 42: Buchholz, B.; Stamm, H. *Chem. Ber.* **1987**, *120*, 1239.
(3) Buchholz, B. Stamm, H. *Isr. J. Chem.* **1986**, *27*, 17.

Scheme I

1, 3: X = O
2, 4: X = NSO$_2$Ph
a: R = Me
b: R = Et

trogen proceeds most easily in the planar transition state of nitrogen inversion. Apart from theoretical considerations (increased ring strain, favorable steric and stereoelectronic conditions), this hypothesis was based[3] on experiments described by Gaertner[5] and on a reactivity comparison[3,6] of aziridines with cyclopropanes possessing similar strength of activation, i.e., similar basicity of their leaving groups. We present now simple experiments that harmonize well with this hypothesis.

A pair of cis–trans isomeric 2,3-disubstituted aziridines was considered suitable as probe for the hypothesis, since the trans isomer will invert faster than the cis isomer, even much faster if the two substituents are rather large. The trans isomer has two indistinguishable invertomers, and in both invertomers the nitrogen pyramid may be flattened for steric reasons. The cis isomer will exist practically exclusively as trans invertomer with a steep pyramid. This preferred trans invertomer of the cis isomer is confronted with a high inversion barrier. Inversion of the cis isomer can therefore be expected to be slower than inversion of the trans isomer.

To avoid possible complications in the intended study by nonsymmetry, identical substituents for positions 2 and 3 should be preferred and, in order to obtain sufficient reactivity, the aziridine should carry an activating group on the nitrogen atom. Pyramidal nitrogen conformation and nitrogen inversion are retained in activated aziridines, although their inversion process is faster than that of aziridine bases.[7] Therefore, we thought it might be informative to make a cis–trans reactivity comparison of such an aziridine pair with the two oxirane counterparts. The two oxiranes[8] and the two aziridines[9] can be expected to react in an S$_N$2 mechanism, provided the activated aziridines do not switch to an SET mechanism.[4,10] The latter possibility is very unlikely in alkoxide attack[3] on sulfonyl-activated aziridines.[4,10]

Fortunately, we found in a paper of Blum et al.[11] the experimental detail that *cis*-stilbene oxide reacts faster (3-h reflux in aqueous acetone) than *trans*-stilbene oxide (48 h) with sodium azide. Therefore we performed simple experiments to find out which isomer of 1 and of 2 reacts faster with sodium methoxide or sodium ethoxide (Scheme I).

(4) Stamm, H.; Sommer, S.; Woderer, A.; Wiesert, W.; Mall, T.; Assithianakis, P. *J. Org. Chem.* **1985**, *50*, 4946.
(5) Gaertner, V. R. *J. Heterocycl. Chem.* **1971**, *8*, 177.
(6) Footnote 20 in ref 4.
(7) Lehn, J. M. *Fortschr. Chem. Forsch.* **1970**, *15*, 313. One of the reviewers proposed we consider an alternative explanation based on a trigonal-planar nitrogen conformation in the ground state of 2. Such a ground state does not harmonize with X-ray results of other *N*-sulfonylaziridines. Compare: Massa, W.; Birkhahn, M.; Landmann, B.; Hoffmann, R. W. *Chem. Ber.* **1983**, *116*, 404 and the literature cited therein.
(8) Parker, R. E.; Isaacs, N. S. *Chem. Rev.* **1959**, *59*, 737.
(9) See, e.g., preceding papers of this series.
(10) Stamm, H.; Assithianakis, P.; Buchholz, B.; Weiss, R. *Tetrahedron Lett.* **1982**, 5021.
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Table I. Base-Catalyzed Alcoholysis of Cis-Trans Pairs of Three-Membered Heterocycles

double expt	reaction conditions	entry	unreacted substrates[a] (%)	sole products[a] (%)
1	7 mmol MeONa 1 mmol 1 50 mL MeOH 3-h reflux	1A 1B	*cis*-1[a] (76) *trans*-1[a] (97)	*threo*-3a[a] (24) *erythro*-3a[a] (3)
2	14 mmol MeONa 1 mmol 1 50 mL MeOH 4.5-day reflux	2A 2B	*cis*-1 (0) *trans*-1 (0)	*threo*-3a (100) *erythro*-3a (100)
3	5 mmol MeONa 1 mmol 2 25 mL MeOH 18 h, 48 ± 2 °C	3A 3B	*cis*-2[a] (42) *trans*-2 (0)	*threo*-4a[a] (58) *erythro*-4a[b] (95)
4	5 mmol EtONa 1 mmol 2 25 mL EtOH 18 h, 48 ± 2 °C	4A 4B	*cis*-2[a] (40) *trans*-2 (0)	*threo*-4b[a] (59) *erythro*-4b[b] (97)
	3 mmol MeONa 3 mmol *cis*-2 40 mL MeOH + 20 mL THF 20.5-h reflux	5	*cis*-2[a] (54)	*threo*-4a[a] (43)

[a]Determined by ¹H NMR analysis. [b]Some material was lost during partitioning between dichloromethane and water.

Table I bears out precisely our expectations. In double experiments 1–4, a cis–trans pair was subjected to identical reaction conditions, resulting in a faster conversion of *cis*-1 and of *trans*-2. We consider this the first direct experimental support of the said hypothesis. To avoid possible complications by secondary reactions, an excess of alkoxide had been used in the double experiments.

Apart from the expected result, the relative reactivity of *cis*-1 and *trans*-1 is interesting. The present and the previous[11] findings that *cis*-1 reacts distinctly faster than *trans*-1 make a recent argument[12] less convincing, as far as it was based on the reactivity comparison between *trans*-1 and unsubstituted oxirane. The reason for the cis–trans difference of 1 is not clear. Steric hindrance may be involved. As space-filling models (Stuart–Briegleb type) show, a rotating phenyl group at position 3 of the ring can be expected to sterically hinder nucleophilic attack on position 2. This effect is possible in *trans*-1 but not in *cis*-1 due to the inability of phenyl in *cis*-1 to adopt the hindering conformation. However, other effects may contribute to the reactivity difference and may even be more important.

Each ring-opening reaction of Table I provided one product not contaminated by its diastereoisomer, quite in accord with an S$_N$2 process. The strong and sharp methoxy signals in the ¹H NMR spectra (250 mHz) of *erythro*-4a and *threo*-4a give as an upper limit of diastereoisomeric contamination less than 0.5%. Assignment of diastereoisomers was based on Walden inversion and was confirmed for *erythro*-3a and *threo*-3a by comparison with literature data.[13,14] The proton coupling H–C–C–H J_{threo} > $J_{erythro}$ is found as expected[15] from the predominance of conformations with an internal hydrogen bridge in 3a, 4a, and 4b.

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(14) The ¹H NMR spectral data of *erythro*- and *threo*-3a compare well with previous data of Hassner and Reuss given for a mixture of both compounds. Their diastereomeric assignment was presented without reasons and has to be interchanged: Hassner A.; Reuss, R. H. *J. Org. Chem.* 1974, 39, 553.
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Experimental Section

General Procedures. ¹H NMR spectra (CDCl₃) were recorded at 250 mHz on a Bruker W 250 spectrometer. Chemical shifts are reported in δ downfield from internal Me₄Si followed in parentheses by peak multiplicity (s, d, t, q, m, m$_c$ = multiplet centered at), coupling constants *J*, and assignment. IR spectra (KBr) were recorded on a Perkin-Elmer 283 spectrometer.

Starting Material. *trans*-Stilbene oxide [*trans*-1: IR 1077 cm⁻¹; ¹H NMR δ 3.89 (s), 7.30–7.44 (m)] was purchased from EMKA, Markgröningen, Germany. *cis*-Stilbene oxide [*cis*-1: IR 1080 cm⁻¹; ¹H NMR δ 4.38 (s), 7.18 (s)] was prepared from *cis*-stilbene and *m*-chloroperbenzoic acid according to a known method.[16] *cis*- and *trans*-1-(phenylsulfonyl)-2,3-diphenylaziridine were prepared from benzenesulfonyl chloride and the respective 2,3-diphenylaziridine according to a known method.[17] The required two 2,3-diphenylaziridines were obtained as described in ref 11 (trans) and in ref 17 (cis).

cis-1-(Phenylsulfonyl)-2,3-diphenylaziridine (*cis*-2): yield 53% (recrystallized from petroleum ether); mp 95–98 °C; IR 1347, 1157 (both SO₂), no absorption at 2800–3000 cm⁻¹; ¹H NMR δ 4.26 (s, 2 aziridine H), 7.00–7.16 (m, Ph, Ph), 7.52–7.69 (m, meta and para H of PhSO₂), 8.07–8.11 (m, ortho H of PhSO₂). Anal. Calcd for C₂₀H₁₇NO₂S: C, 71.64; H, 5.07; N, 4.18. Found: C, 71.92; H, 5.27; N, 4.10.

trans-1-(Phenylsulfonyl)-2,3-diphenylaziridine (*trans*-2): yield 71% (recrystallized from petroleum ether); mp 119–122 °C; IR 1328, 1171, 1164 (all SO₂), no absorption at 2800–3000 cm⁻¹; ¹H NMR δ 3.53 (s, 2 aziridine H), 7.33–7.44 (m, Ph, Ph, meta H of PhSO₂), 7.50–7.57 (m, para H of PhSO₂), 7.73–7.77 (m, ortho H of PhSO₂). Anal. Calcd for C₂₀H₁₇NO₂S: C, 71.64; H, 5.07; N, 4.18. Found: C, 71.36; H, 4.96; N, 4.33.

Reactions of Table I. All experiments were performed with continuous stirring. 1 or 2 was added to the solution of sodium in the listed alcohol (with THF added in entry 5). The mixture was brought to the temperature given in Table I. The reactions were finished by evaporation in a rotatory evaporator at bath temperatures below the reaction temperature. The residue was taken up in dichloromethane, washed with water to neutral reaction, and evaporated again. In entries, 2A, 2B, 3B, and 4B, the obtained residue consisted only of the respective product. In entries 1A, 1B, 3A, and 4A, the weighed residue was analyzed by means of its ¹H NMR spectrum. In entry 4A, part of the residue was subsequently subjected to preparative TLC (Merck 60F₂₅₄ plates, 2 mm thick, 20 cm × 20 cm, dichloromethane). The lower zone (visible in UV light) was scraped out and extracted with hot ethyl acetate. Evaporation of the clear extract provided pure *threo*-4b. In entry 5, the residue was chromatographed (silica gel, 0.063–0.2 mm, Merck, 1.5 cm × 60 cm). Elution with dichloromethane provided 510 mg (54%) of *cis*-2 followed by 454 mg (43%) of *threo*-4a.

threo-2-Methoxy-1,2-diphenylethanol (*threo*-3a): mp 51–52 °C (lit.[13] mp 53–54 °C); IR 3480 (OH), 1101 (C–O), 1062 cm⁻¹ (C–O); ¹H NMR δ 3.31 (s, MeO) 3.53 (s br, OH), 4.12 (d, *J* = 8.4 Hz, CHOR), 4.65 (d, *J* = 8.4 Hz, CHOH), 6.97–7.07 (m, 4 ortho H of 2 Ph), 7.14–7.26 (m, 4 meta H and 2 para H of 2 Ph).

erythro-2-Methoxy-1,2-diphenylethanol (*erythro*-3a): mp 100–103 °C (lit.[13] mp 101–102 °C); IR 3440 (OH), 1104 (C–O), 1088 (C–O), 1062 cm⁻¹ (C–O); ¹H NMR δ 2.36 (d, *J* = 4.0 Hz, OH), 3.23 (s, MeO), 4.35 (d, *J* = 5.4 Hz, CHOR), 4.90 (m$_c$, CHOH), 7.11–7.21 (m, 4 ortho H of 2 Ph), 7.21–7.32 (m, 4 meta H and 2 para H of 2 Ph).

threo-N-(2-Methoxy-1,2-diphenylethyl)benzenesulfonamide (*threo*-4a): mp 118–120 °C; IR 3340 (NH), 1328 (SO₂), 1157 (SO₂), 1110 (C–O), 1090 cm⁻¹ (C–O); ¹H NMR δ 3.18 (s, MeO), 4.18 (d, *J* = 7.2 Hz, CHOR), 4.36 (dd, J_{HCCH} = 7.2 Hz, J_{HCNH} = 3.6 Hz, CHN), 5.74 (d br, *J* = 3.4 Hz, NH), 6.88–7.45 (m, 13 aromatic H), 7.45–7.53 (m, 2 ortho H of PhSO₅). Anal. Calcd for C₂₁H₂₁NO₃S: C, 68.64; H, 5.76; N, 3.81. Found: C, 68.25; H, 5.74; N, 3.88.

erythro-N-(2-Methoxy-1,2-diphenylethyl)benzenesulfonamide (*erythro*-4a): mp 125–127 °C; IR 3350 (NH), 3290 (NH),

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1332 (SO$_2$), 1170 (SO$_2$), 1094 cm^{-1} (C–O); ^{1}H NMR δ 3.18 (s, MeO), 4.51 (d, *J* = 4.1 Hz, CHOR), 4.55 (dd, *J*$_{HCCH}$ = 4.1 Hz, *J*$_{HCNH}$ = 8.5 Hz, CHN), 5.77 (d, *J* = 8.5 Hz, NH), 6.70–6.75 (m, 2 ortho H of Ph), 6.77–6.84 (m, 2 ortho H of Ph), 6.89–7.08 (m, 3 aromatic H, 7.09–7.29 (m, 5 aromatic H), 7.31–7.41 (m, 1 aromatic H), 7.54–7.62 (m 2 ortho H of PhSO$_2$). Anal. Calcd for C$_{21}$H$_{21}$NO$_3$S: C, 68.64; H, 5.76; N, 3.81. Found: C, 68.53; H, 5.84; N, 4.08.

threo-*N*-(2-Ethoxy-1,2-diphenylethyl)benzenesulfonamide (*threo*-4b): mp 93–95 °C; IR 3300 (NH), 1329 (SO$_2$), 1166 (SO$_2$), 1093 (C–O), 1076 cm^{-1} (C–O); ^{1}H NMR: δ NMR: δ 1.13 (t, *J* = 7.0 Hz, OCMe), 3.23 (m$_c$, 1 H of OCH$_2$), 3.36 (m$_c$, 1 H of OCH$_2$), 4.27 (d, *J* = 6.6 Hz, C*H*OR), 4.35 (dd, *J*$_{HCCH}$ = 6.6 Hz, *J*$_{HCNH}$ = 3.8 Hz, CHN), 5.74 (d, *J* = 3.7 Hz, NH), 6.93–7.27 (m, 12 aromatic H), 7.34–7.44 (m, 1 aromatic H), 7.45–7.51 (m, 2 ortho H of PhSO$_2$). Anal. Calcd for C$_{22}$H$_{23}$NO$_3$S: C, 69.26; H, 6.08; H, 3.67. Found: C, .69.02; H, 6.26; N, 3.23.

erythro-*N*-(2-Ethoxy-1,2-diphenylethyl)benzenesulfon-amide (*erythro*-4b): mp 113–115 °C; IR 3290 (NH), 1327 (SO$_2$), 1168 (SO$_2$), 1092 (C–O), 1077 cm^{-1} (C–O); ^{1}H NMR δ 1.14 (t, *J* = 7.0 Hz, OCMe), 3.23 (m$_c$, 1 H of OCH$_2$), 3.39 (m$_c$, 1 H of OCH$_2$), 4.53 (dd, *J*$_{HCCH}$ = 4.0 Hz, *J*$_{HCNH}$ = 8.7 Hz, CHN), 4.62 (d, *J* = 4.0 Hz, CHOR), 5.63 (d, *J* = 8.8 Hz, NH), 6.72–6.79 (m, 2 ortho H of Ph), 6.79–6.86 (m, 2 ortho H of Ph), 6.90–7.31 (m, 8 aromatic H), 7.32–7.42 (m, 1 aromatic H), 7.56–7.63 (m, 2 ortho H of PhSO$_2$). Anal. Calcd for C$_{22}$H$_{23}$NO$_3$S: C, 69.26; H, 6.08; N, 3.67. Found: C, 69.24; H, 6.14; N, 3.63.

Micellar Catalysis of Organic Reactions. 20.[†] Kinetic Studies of the Hydrolysis of Aspirin Derivatives in Micelles

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Introduction

The hydrolysis of 2-(acetyloxy)benzoic acid (aspirin) (1) has been studied extensively because of its pharmaceutical importance and also because of its chemical interest.[1] The pH–rate profile[2] shows a plateau between pH 5 and 9, and above pH 9 the rate increases as the pH is increased. In the plateau region, it has been concluded that the mechanism of reaction involves intramolecular general base catalysis by the ionized carboxy group.[1] At higher pH (>9), hydrolysis by the normal B$_{AC}$2 mechanism (hydroxide attack at the carbonyl carbon of the ester) is observed.

Most of the previous studies of aspirin hydrolysis have been carried out in aqueous solution, although this is possibly not the most appropriate medium from which to draw conclusions regarding the stability of aspirin in biological systems. Studies in the presence of biological membranes would be of considerably more relevance. Micelles have long been recognized to be simplistic models of biological membranes.[3,4] Thus, it follows that a study of the hydrolysis in the presence of micelles may be a better model than studies in water from which to draw

conclusions concerning the stability of aspirin in biological systems.

Previous work[5] on the hydrolysis of aspirin has shown that in the presence of micelles of cetyltrimethyl-ammonium bromide (CTAB), intramolecular general base catalysis at pH 6–8 is less efficient than in water, whereas the normal B$_{AC}$2 hydrolysis (at pH >9) is slightly catalyzed. Computer simulation[6] of the variation of the observed rate of reaction (k_ψ) with surfactant concentration at pH 12, however, has shown that for the best fit the second-order rate constant in the micellar pseudophase (k_2^m) is approximately 100 times smaller than that for reaction in water. The slight observed catalysis is due to concentration of the substrate within the micellar pseudophase for which the binding constant, K_s, is 190–340 M^{-1}, depending on the hydroxide concentration. It was also found that the second-ordder rate constant (k_2^m) calculated from computer simulation varied with hydroxide concentration (k_2^m) = 0.09 – 0.147 M^{-1} m^{-1}). Constant values of K_s and k_2^m, independent of hydroxide concentration, were obtained by a more recent iterative calcualtion method[7] in which the value of β, the fraction of micellar head groups neutralized, was allowed to vary with the hydroxide and surfactant concentrations.

One of the important factors for reactions in micelles is the orientation of the substrate molecule in the micellar pseudophase and the resultant location of the reaction center. For this reason, we chose to study the hydrolysis of some derivatives (2, 3) of aspirin (1) containing hydrophobic chains.
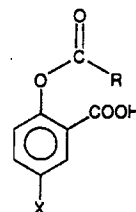
1, X = H; R = CH$_3$
2, X = H; R = C$_7$H$_{15}$
3, X = C$_8$H$_{17}$; R = CH$_3$

It was hoped that the orientation of the substrate in the micelle would vary as the site of the hydrophobic chain was varied and the effect of this on the kinetics of hydrolysis has been studied. Some support for the conclusions based on the kinetic results has been obtained from NMR studies[8] and the observation of viscoelasticity in some systems.[9]

Results and Discussion

Reaction in Basic Solution (pH 12.0). Weak catalysis was observed for all compounds ranging from 2.2 (compound 2) to 6.3 (compound 3). In all cases, the rate–[CTAB] profile exhibited a maximum corresponding to complete solubilization of the substrate in the micellar

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